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**Title of the Invention: Novel Polyester-Based Hot Melt Adhesive**

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(54) Title of the Invention: **Novel Polyester-Based Hot Melt Adhesive**

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**SPECIFICATION**

**1. Title of the Invention**

Novel Polyester-Based Hot Melt Adhesive

**2. Claims**

A novel polyester-based hot melt adhesive characterized by comprising a homogeneous mixture composed of 50-97% by weight of a saturated polyester (I) in which the acid component is composed of at least two acids, at least one of the components is aromatic, the melt index at 140°C is 50-400 g/10 min, and the softening temperature is 80-130°C; and 50-3% by weight of a vinyl polymer (II) in which the melt index at 190°C is 0.5-20 g/10 min and the softening temperature is 70-160°C.

### **3. Detailed Description of the Invention**

The present invention relates to a novel polyester-based hot melt adhesive. More specifically, it relates to a polyester-based hot melt adhesive with excellent adhesiveness and durability that is appropriate for fiber materials.

Examples of fiber materials used with hot melt adhesives, and examples of applications for such materials include adhesive padding in which an adhesive is applied beforehand to a fabric or nonwoven fabric, temporary bonding to assist in sewing, adhesive sewing applications, seals, and permanent adhesives for badges. Polyethylene, ethylene-vinyl acetate copolymers, ethylene-acrylate copolymers, and polyamide-based resins are used as the hot melt adhesives for these applications. However, higher performance has come to be demanded, especially of permanent adhesive paddings and adhesive sewing. Conventional adhesives are also incapable of ensuring adequate adhesive strength because the main fiber material on which the adhesives are used is polyester. This trend is especially conspicuous in adhesive padding, which has recently come to be processed in far larger quantities. Conventional adhesives are clearly lacking in durability. There has consequently been an increase recently in the consumption of polyester-based hot melt adhesives that have excellent adhesiveness and durability for polyester fibers.

A two-step process is often employed for hot melt adhesives used with textiles whereby the hot melt adhesive is applied to part or all of the fabric surface in the form of a powder, film, filament, net, staple, dispersion, paste, or the like; bonded to the fabric surface by heating; and heated and bonded by pressure from an iron or press to another fabric as necessary.

Hot melt adhesives for textiles therefore must have different performance from ordinary hot melt adhesives used for wood, adhesive tape, and paper processing.

The melt viscosity of ordinary hot melt adhesives is preferably kept low to improve the applicability to the material to be bonded and to raise the adhesive strength. The melt viscosity of hot melt adhesives used with textiles, however, must be higher so that the adhesive does not penetrate the fiber structure when hot pressed. Deep penetration of the fiber structure by the adhesive roughens and hardens the feel of the fabric, causes staining by the adhesive, and greatly decreases the adhesive strength.

On the other hand, the hot melt adhesives used with textiles must be able to adhere at a relatively low temperature. Even though the heat resistance of some textiles such as rayon, cotton, and linen reaches 180-200°C, that of polypropylene fibers, polyamide fibers, polyester fibers, wool, and silk is low at 140-170°C, and that of acetate fibers and acrylic fibers is extremely low at 80-120°C. This means that discoloration of the fibers, changes in quality, changes in shape, and the like are unavoidable, especially in the case of ordinary polyamide and polyester fibers. The temperature during pressing is 140-170°C and the duration of pressing is about 5-30 seconds. The hot melt adhesive therefore must be easy to use under such conditions. Adhesives capable of adhering at lower temperatures are desired to expand the range of textile materials on which they can be used. Thus, hot melt adhesives for textiles must be able to adhere at a relatively low temperature and must have as high a melt viscosity as possible.

Polyester resin, the main polyester-based hot melt adhesive currently in use, is produced by using a special reactor in which a high vacuum is maintained in order to raise the melt viscosity, performing a condensation reaction at a high temperature of 200°C or higher under an extremely high vacuum of 1 mmHg or less, and continuing the reaction until a resin with sufficiently high viscosity (a melt viscosity of approximately 100,000 cps or more at 200°C) is obtained. However, the special reactor in which such a high vacuum is maintained is dangerous to handle, the production equipment is expensive, and production costs are high.

Taking a completely different viewpoint, the present inventors perfected the present invention upon discovering that the desired hot melt adhesive can be obtained by using a relatively low-molecular-weight polyester without using a special high-vacuum reactor.

In summary, the present invention is "a novel polyester-based hot melt adhesive characterized by comprising a homogeneous mixture composed of 50-97% by weight of a saturated polyester (I) in which the acid component is composed of at least two acids, at least one of the components is aromatic, the melt index at 140°C is 50-400 g/10 min, and the softening temperature is 80-180°C<sup>1</sup>; and 3-50% by weight of a vinyl polymer (II) in which the melt index at 190°C is 0.5-20 g/10 min and the softening temperature is 70-160°C."

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<sup>1</sup> Translator's note: This range is indicated as being "80-130°C" in the claims.

The carboxylic acid component that constitutes the polyester (I) used in the present invention is composed of (i) two or more aromatic carboxylic acid components or (ii) an aromatic dicarboxylic acid component and an aliphatic dicarboxylic acid component. Examples of the aromatic dicarboxylic acid component include terephthalic acid, isophthalic acid, 2,6-naphthalenedicarboxylic acid, and lower alkyl esters of these. Examples of aliphatic dicarboxylic acid components include adipic acid, azelaic acid, sebacic acid, and dodecanedioic acid. Examples of the glycol component that constitutes the polyester (I) include ethylene glycol, trimethylene glycol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol, neopentyl glycol, and other such alkylene glycols and polyoxyalkylene glycols. Various polyester properties are obtained by combining the acid component and glycol component. A combination is selected and the degree of polymerization is also adjusted in the present invention so that the melt index is 50-400 g/10 min, preferably 70-300 g/10 min, and the softening temperature is 80-180°C, preferably 100-150°C. Polyester (I) can be synthesized easily by using an ordinary reactor equipped with vacuum distillation equipment and maintained at a maximum degree of vacuum of about 20 mmHg.

The vinyl polymer (II) is a homopolymer or copolymer of ethylene, propylene, styrene, vinyl acetate, alkyl (having 1-4 carbon atoms in the alkyl groups) esters of acrylic acid and methacrylic acid, acrylonitrile, maleic anhydride, and the like. Polyethylene and ethylene-vinyl acetate copolymers are preferred. Of course, the vinyl polymer (II) used in the present invention must have a melt index of 0.5-20 g/10 min, preferably 1-10 g/10 min, and a softening temperature of 70-160°C, preferably 80-140°C.

When the melt index of the vinyl polymer is lower than 0.5, it becomes extremely difficult to mix with the polyester (I) because its melt viscosity is too high, which is impractical. When the melt index is higher than 20 g/10 min, the melt viscosity of the adhesive cannot be raised because the melt viscosity [of the vinyl polymer] is too low. When the content of the vinyl polymer is raised to compensate for this, either there is no effect or the durability of the adhesive drops in the extreme.

The amount of the vinyl copolymer (II) versus the polyester resin (I) in the adhesive of the present invention is adjusted so that there is a homogeneous mixture of 3-50% by weight based on the mixture of the two.

When there is less than 3% by weight of the vinyl polymer (II), the melt viscosity of the adhesive rises. When there is more than 50% by weight, the water resistance of the adhesive and its solvent resistance, adhesiveness to polyester fibers, and bonding durability decline. The preferred content of the vinyl polymer (II) is 5-20% by weight.

The method of mixing the polyester (I) and vinyl polymer (II) is not particularly restricted. A powder, pellets, dispersion, emulsion, and the like of the polyester (I) is usually simply mixed until homogeneous with a powder, pellets, dispersion, emulsion, and the like of the vinyl polymer (II), or the vinyl polymer (II) is added to a molten solution of the polyester (I) and kneaded until homogeneous. A solvent such as propyl acetate, hexyl acetate, benzyl acetate, trichloroethylene, 1,1,2-trichloroethane, or tetrachloroethylene may also be used if necessary and removed after having dispersed the vinyl polymer (II) homogeneously in the polyester (I).

The melt index of the adhesive of the present invention is far higher than that of the polyester (I) alone. This resolves problems such as excessive penetration of the fiber structure by the adhesive during pressing and other types of processing, the resulting inadequate adhesive strength, and staining of the surface of the fabric.

The adhesive of the present invention can be provided for bonding in the form of a powder, film, or yarn, or as a dispersion dispersed in water, as in the past. Molten film formation is preferred for film formation. Melt extrusion is carried out using an ordinary extruder to obtain the film. The hot melt adhesive film thus obtained is bonded by being placed between fabrics of the same or different types, and heating and melting the film with the aid of an iron, hot press, or hot roller. To produce a powder, the adhesive is crushed and pulverized in a crusher after having been cooled with a coolant such as liquid nitrogen. The powdered adhesive is applied using a machine such as a dotting machine or scatter machine. A dispersion is produced by high-speed stirring in a pressurized vessel, and the dispersion is applied using a spraying apparatus or a knife coater.

Examples of the fiber materials that can be bonded with such adhesives include synthetic fibers such as polyester, polyamide, and acrylic; natural fibers such as wool and cotton; knit fabrics made from mixtures of these; nonwoven fabrics; and all fiber products.

The present invention permits the adhesive to be used with fibers such as polyester resins with a melt index at 140°C of 50-300 g/10 min, on which hot melt adhesives for textiles could not be used in the past because the adhesive penetrated the fiber structure during hot pressing. As a result, the polyester resin can be produced relatively easily without using a special high-vacuum reactor, the equipment is greatly simplified, and energy can be saved.

Antioxidants, slip agents, antiblocking agents, tackifiers, and the like can also be added in appropriate quantities as needed to the adhesive of the present invention.

The present invention is explained concretely below through reference and working examples. The present invention, however, is not limited to these working examples alone. Below, "parts" and "%" are based on weight unless specifically stated otherwise.

### **Reference Example**

#### **[I] Synthesis of polyester (I)**

291 g (1.5 mol) of dimethyl terephthalate, 405 g (4.5 mol) of 1,4-butanediol, and 0.5 g of potassium titanyl oxalate were placed in a reactor, heated to 140-200°C in a nitrogen stream, and transesterified by a demethanolization reaction. Next, 345 g (1.5 mol) of dodecanedioic acid was added, heated to 240°C, and polycondensed for 4 hours under 23 mmHg reduced pressure.

Polyester (a) was synthesized.

The polyester (a) thus obtained had a softening temperature of 123°C and a melt index of 150 g/10 min. Copolymers (b)-(d) of the polymer compositions shown in Table 1 were synthesized in the same way.

Table 1

Polyester	Acid component	Molar ratio	Glycol component	Softening point (°C)	Melt index 140°C (g/10 min)
I-a	Terephthalic acid Dodecanoic acid	50 50	1,4-Butanediol	123	150
I-b	Terephthalic acid Isophthalic acid	40 60	Ethylene glycol	135	70
I-c	Isophthalic acid Adipic acid	60 40	1,5-Pentanediol	96	350
I-d	Terephthalic acid Sebatic acid	40 60	1,3-Butanediol	108	210

**[II] Vinyl polymer admixed**

Table 2

Symbol	Vinyl polymer	Softening temperature (°C)	Melt index 190°C (g/10 min)
II-a	High-density polyethylene	120	10
II-b	Ethylene-vinyl acetate copolymer resin	80	1
II-c	Ethylene- $\alpha$ -olefin copolymer resin	105	15
II-d	Polypropylene	140	5

**Working Example 1**

The polyester obtained in Reference Example [I] and various types of vinyl polymers (B) in [II] were melt kneaded and cooled, and a finely powdered adhesive was produced by cold crushing. The adhesive was bonded in a proportion of 20 g/m<sup>2</sup> to spun polyester padding by powder coating to produce a padding product.

The resin on the spun polyester padding was then melt bonded by heating for 30 seconds at 140°C, and adhesive padding was produced. The adhesive padding obtained was press bonded

to a polyester/cotton = 65/35 mixed spun broadcloth at a temperature of 150°C and a pressure of 0.30 kg/cm<sup>2</sup>, with 1 second of presteaming and 9 seconds of pressing.

The 180° peel strength was determined (unit: kg/25 mm) at a pull rate of 100 mm/min using an Autograph P-100 (made by Shimadzu) in measurement of the adhesive strength. Dry cleaning and laundering in water were also conducted according to JIS L-1089, and the adhesive strength was measured thereafter. The results are shown in the table below.

A 180° peel strength of 1.2 kg/25 mm at the start, after dry cleaning, and after washing was taken to be a passing adhesive strength.

Table 3

Adhesive	Working examples							Comparative examples			
	1	2	3	4	5	6	7	1	2	3	4
<b>Composition</b>											
Polyester 100 parts	I-a	I-a	I-a	I-a	I-b	I-c	I-d	I-a	I-d	I-b	I-a
Vinyl polymer	II-a	II-a	II-a	II-b	II-c	II-d	II-d				II-a
No. of parts of vinyl polymer	5	15	30	40	15	15	15				60
<b>Properties</b>											
140°C Melt index (g/10 min)	10	5	4	3.5	7	8	6	150	210	70	3
Softening temperature (°C)	122	121	121	86	110	100	112	123	108	135	120
<b>Adhesive strength (kg/25 mm)</b>											
Starting	1.66	1.81	1.79	1.76	1.94	1.90	1.80	*	*	1.24	1.31
After dry cleaning	1.48	1.70	1.55	1.52	1.73	1.69	1.63	—	—	1.02	0.93
After washing	1.36	1.44	1.36	1.33	1.47	1.47	1.44	—	—	0.86	0.75

\*Could not be used because of extreme penetration of the adhesive.

## Working Example 2

Polyester (I-a) was finely pulverized by cold crushing in liquid nitrogen, and a powdered polyester resin was produced. An ethylene-vinyl acetate copolymer resin of II-b with a softening temperature of 80°C and a melt index (190°C) of 1 g/10 min was finely pulverized by cold crushing in the same way. The two were homogeneously mixed in a ratio of I-a/II-b = 8/2 using a Banbury mixer immediately before use, and an adhesive was prepared. In the production of padding, the adhesive was applied in a proportion of 18 g/m<sup>2</sup> by powder coating on spun polyester padding and heated for 10 seconds at 160°C, and the adhesive was bonded to the spun polyester padding to produce adhesive padding.

The adhesive padding thus obtained was press bonded to polyester/cotton = 65/35 mixed spun broadcloth at a temperature of 150°C and a pressure of 0.30 kg/cm<sup>2</sup>, with 1 second of presteaming and 6 seconds of pressing. The adhesive did not penetrate the fiber structure at all in the bonded padding. The starting adhesive strength was 1.86 kg/25 mm, the strength after dry cleaning was 1.72 kg/25 mm, and the strength after washing was 1.46 kg/25 mm.

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